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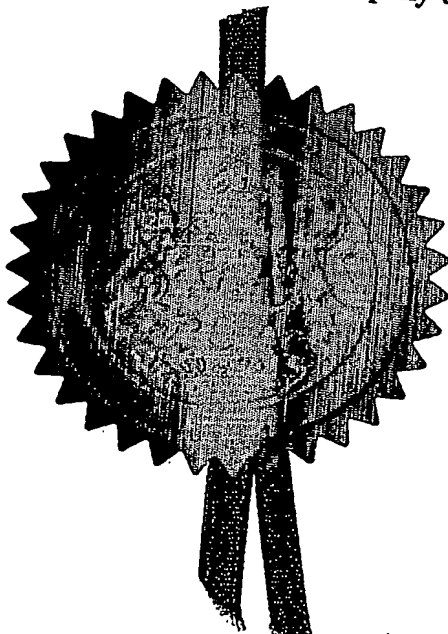
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04SEP02 E745818-1 D02992
P01/7700 0.00-0220551.6

2. Patent application number
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0220551.6

- 4 SEP 2002

3. Full name, address and postcode of the or of each applicant (underline all surnames)

CLARIANT INTERNATIONAL LTD
Rothausstrasse 61
CH-4132 Muttenz

Patents ADP number (if you know it)

6971634001

If the applicant is a corporate body, give the country/state of its incorporation

Switzerland

4. Title of the invention

EQUAL DENSITY PELLETS OR MICRO PELLETS

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

CLARIANT UK LTD
Attn. Ms. Anne Plowden
Calverley Lane
Horsforth/Leeds, LS18 4RP

Patents ADP number (if you know it)

7156086001

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Priority application number
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Date of filing
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Number of earlier application

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Yes

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Description

13 ✓

Claim(s)

2 ✓

Abstract

1 ✓

Drawing (s)

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Request for preliminary examination and search (Patents Form 9/77)

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Request for substantive examination (Patents Form 10/77)

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11.

I/We request the grant of a patent on the basis of this application.

Signature

Clariant International Ltd

Date

September 4, 2002

Florian Bielefeldt

Patent Attorney

C. Heusler

Christel Heusler

Patent Administrator

12. Name and daytime telephone number of person to contact in the United Kingdom

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PATENTS FORM 1/77

DUPLICATE

Equal density pellets or micro pellets**Background of the invention**

5 For coloring polymers, the plastic manufacturers prefer to purchase *ready to use products* from color manufacturers instead of working with the basic colorants (pigments and dyes) which require a comprehensive formulation and color matching expertise in order to achieve the requested color and the specific properties of the polymers.

10

The most common product forms sold by the color manufacturers are pre-colored resins or color concentrates. Color concentrates contain a high concentration of pigments or dyes in a carrier system. Such color concentrates are mixed by the plastic manufacturer with uncolored polymer and are then manufactured into semifinished products or final
15 products. The color concentrates are commonly used in pelletized form (also known as *masterbatches*) or liquid form (also known as liquid color).

The manufacturers of pre-colored resins use virgin resin and add colorants and functional additives. The final product is a pellet which is ready for processing.

20 Compared to other methods of coloring resin, the use of pre-colored resin is a rather expensive manufacturing practice. Pre-colored resin is much more expensive than uncolored resin plus a color concentrate.

The manufacturers of color concentrates frequently have to produce small quantities of
25 masterbatches (usually 10 to 50 kg) within short lead times due to the fact, that the plastic manufacturers want to keep only minimal stock of color concentrates in case a color becomes suddenly obsolete.

The color concentrates, either in liquid form or in the pellet form (masterbatch) have to
30 be mixed with the uncolored resin in a particular let-down ratio in order to achieve the desired color. The mixing can be done either by manual mixing, or, more commonly, by a batch-blend process using automated dosing equipment.

Liquid colors are normally pumped directly into the feed throat of the processing machine (e.g. an injection molding machine, a blow molding machine, a melt spinning system or the like) by using a small peristaltic pump. A problem related with liquid color concentrates is, that the liquid carrier can have a negative impact on the physical properties of the polymer. Further disadvantage of liquid colors compared to masterbatches are a more difficult handling, the cleaning of the machine between color changes, and the processing of the liquid color. However, the coloring costs with liquid colors are lower compared to conventional masterbatches.

- 10 For most plastic manufacturers, the use of masterbatches is the preferred method for coloring of plastics. Ease of handling and low costs compared to pre-colored resins are the main advantages for these products. Unlike liquid colors, the compatibility of masterbatches with the resin is excellent, since the carrier material of the masterbatch is in most cases identical with the resin.

15

Masterbatches are commonly supplied in pellet or in micro-pellet form. There are few ways to produce a masterbatch. The first and most common way used by the masterbatch manufacturers is a so called one step process. During this process, pigments (or dyes), functional additives and the polymer carrier are mixed, the mixture is then processed through a dispersion unit, e.g. a co-rotating twin screw extruder.

The color concentrates (masterbatches) can also be obtained in a so called two step process, wherein in a first step, the masterbatch manufacturers produce a Single Pigment Concentrate (SPC). SPC is a masterbatch preparation containing only one pigment or dye (generally in very high concentration) fully dispersed in a resin carrier optionally comprising further additives. This first process step is very similar to the one step process.

In a second step, different SPCs are mixed together in order to achieve the required color specifications of the customer and to get the tailor made masterbatch. To reduce the pigment concentration, the SPCs are mixed together with some carrier resin. Since SPCs are already fully dispersed, only a mixing process is necessary. This can be performed for example with a single screw or twin screw extruder.

Compared to the one step process, the two step process leads to a much higher flexibility in the production of tailor made masterbatches and is also more cost-effective for the production of small production batches (low cleaning costs of the machines, higher output rate compared to the one step process). This is increasingly important
5 since the color lifetime in the plastics business is becoming shorter and the average lot size is becoming smaller.

The main disadvantage of this process is that it requires a second extrusion which raises the production cost of the resulting tailor-made masterbatches and which can affect the
10 mechanical properties of the carrier polymer and therefore also the mechanical properties of the final product.

A possibility to avoid the second extrusion is to separately add each SPC to the resin during processing. This requires a dosing equipment for each SPC. The dosing units are
15 mounted directly onto the feed throat of the processing equipment (e.g. an injection molding machine, a blow molding machine, a melt spinning system or the like). This system, a so-called multi dosing system, is not commonly used because it is very sophisticated and expensive.

20 The direct feeding of pre-mixed SPCs in a single dosing system in order to avoid the second extrusion, leads to a separation or de-mixing of the different components due to the difference in bulk density, due to the difference in electrostatic properties of the different components and due to the difference in form and size of the pellets.

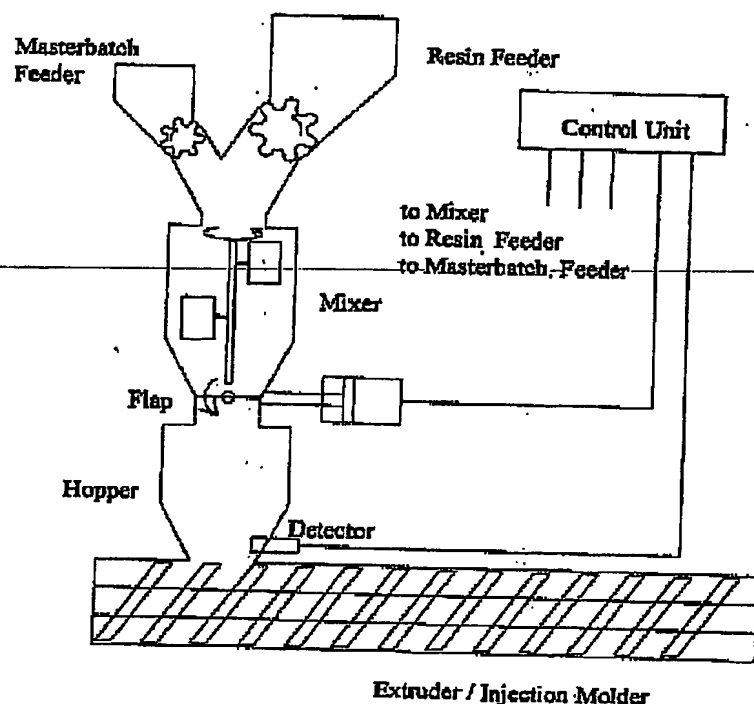
The separation or de-mixing can take place during the following processing steps:

- 25
- loading of the pre-mixed SPCs in transport or storage containers
 - transport of the pre-mixed SPCs in transport containers
 - pneumatic conveying from the storage containers into the dosing unit
 - feeding of the injection molding or extrusion machine under the influence of the equipment's vibration.

30 The separation leads to inconsistent and inhomogeneous color of the final products.

The separation in a single dosing system can be avoided by using an automated dosing equipment, i.e. a volumetric or gravimetric feeder. The dosing unit is mounted directly onto the feed-throat of the processing equipment (e.g. an injection molding machine, a blow molding machine, a melt spinning system or the like). The resin is filled either manually into a hopper attached to the dosing unit or pneumatically conveyed from silos or storage containers directly into the dosing unit (see Figure 1 below).

Figure 1 : Example of a volumetric feeding unit



10

Another approach to avoid separation of components is disclosed in JP62009915, wherein a plastic molding method is described which provides for plastic pellets with different load or properties which do not separate in the feed. JP62009915 proposes to use larger pellets if the specific gravity (density) and/or antistatic properties of the material are low; or to use smaller pellets if the specific gravity (density) and/or the antistatic properties are high. The process of JP62009915 therefore compensates the different densities of the material, as a first parameter, by adapting the size of the pellets and, additionally, takes into account the dielectricity of the material as a second parameter.

Summary of the Invention

The present invention provides a method for preventing the separation of at least two different Single Pigment Concentrates (SPC) and/or Additive Concentrates in pellet or micro pellet form.

- 5 Pellets or micro pellets according to the invention are characterised in that one or more Single Pigment Concentrates (SPCs) and of one or more Additive Concentrates with at least two different chemical compositions,
or of two or more Single Pigment Concentrates (SPCs) with at least two different chemical compositions,
10 or of two or more Additive Concentrates with at least two different chemical compositions,
with substantially equal density are provided.

~~The invention thereby enables the preparation of more cost effective tailor-made~~

- 15 masterbatches with SPC and/or Additive Concentrates by avoiding the multi-extrusion.
The invention further provides a process with higher flexibility and reactivity in the production of tailor made masterbatches.

In a further aspect, the invention provides:

- 20 ▪ a method of coloring plastic by using a blend (or mixture) of SPCs of at least two components.
 ▪ a method of bringing properties to the plastic by using a blend (or mixture) of Additive Concentrates of at least two components.
 ▪ a method of coloring and bringing properties to the plastic by using a blend of
25 SPCs and Additive Concentrates of at least two components.

- The Additive Concentrates are defined as preparations containing at least one active ingredient dispersed in a resin carrier, wherein the active ingredient is selected from the group consisting of anti-blocking, anti-fogging, anti-microbial, antioxidant, anti-slipping, anti-static or cleaning agents, compatibilizers, conductive agents, corrosion
30 inhibitors, de-nesting agents, drying agents, fillers, flame retardants, foaming agents, infrared agents, laser marker agents, lubricants, matting agents, nucleating agents,

opacifiers, optical brightener, phosphorescent agents, photo-degradable agents, processing aids and/or UV stabilisers.

5 The Single Pigment Concentrates (SPCs) are defined as preparations containing only one pigment or dye dispersed in a carrier resin optionally comprising additives (see the list above). A SPC contains 20 to 80 % by weight of a pigment or a dye and 80 to 20% by weight of a carrier polymer. Pigments are inorganic or organic pigments.

10 The carrier polymer is a thermoplastic polymer or a blend of thermoplastic polymers selected from polyolefins, polyesters, polyamides, polyacrylates, polycarbonates, polyurethanes, polystyrenes and copolymers thereof. It is required, that the carrier polymer for the masterbatch is compatible with the virgin polymer used for the final product.

15 The SPCs and Additive Concentrates are provided in pellet or in micro pellet form. The micro pellet form is preferred for an improved color uniformity of the final product due to the smaller size and better statistical distribution of the micro pellets. The size of the pellets is from larger than 2.0 mm to 4.0 mm; the size of the micro pellets is from about 0.5 mm to 2.0 mm.

20

This technology can be easily introduced in a variety of applications for example in injection molding, blow molding, extrusion, spin dyeing. The color consistency is insured from the beginning to the final product since the mixture of the components stays homogeneous over the time.

25

The main characteristics of the components (micro pellets or pellets) according to the invention are:

- controlled density (specific weight) of the components (micro pellets or pellets)
- controlled size and shape of the components (micro pellets or pellets)
- 30 ▪ controlled electrostatic properties of the components (micro pellets or pellets)

Masterbatches according to the invention consist of micro pellets or pellets of different chemical composition but with substantially equal density.

The density of the pellets or micro pellets is from 0.5 to 1.5 g/cm³ and preferably from 0.6 to 1.1 g/cm³.

- 5 Preferred masterbatches according to the invention consist of micro pellets or pellets of different chemical composition but with substantially equal density and substantially equal size and shape.

- 10 Most preferred masterbatches according to the invention consist of micro pellets or pellets of different chemical composition but with substantially equal density, substantially equal size and shape and substantially equal electrostatic properties.

- Micro pellets or pellets of different chemical composition but with substantially equal density according to the invention are obtained by adjusting the pigment content, by
15 adjusting the filler content and/or by adding a blowing agent to produce a hollow pellet.

- Micro pellets or pellets of different chemical composition but with substantially equal density and substantially equal size and shape are obtained by adjusting the pigment content, adjusting the filler content and/or adding a blowing agent to produce a hollow
20 pellet and subjecting the blend to the same production and shaping process.

- To prevent the separation of the mixture due to the electrostatic charge of the components, an antistatic additive (for example stearates or lauryl amides) is added in the blend to obtain the most preferred micro-pellets or pellets of different chemical
25 composition but with substantially equal density, substantially equal size and shape and substantially equal electrostatic properties.

- The invention therefore enables the production of SPCs and Additive Concentrates of different chemical composition with the same specific weight (density) of the pellets or
30 micro-pellets.

The adjustment of the parameters of the different SPCs and Additive Concentrates can be achieved as follows:

- 1) Take the SPC or Additive Concentrate with the composition of lower specific weight as reference and reduce the specific weight of all other SPCs and Additive Concentrates to the specific weight of the reference by:
- reducing the concentration of pigment (respectively additive) of the SPCs or Additive Concentrates
 - or by adding a blowing agent in order to form "hollow" pellets or micro pellets.
- 2) Take the SPC or Additive Concentrate with the composition of higher specific weight as reference and increase the specific weight of all other SPCs and Additive Concentrates to the specific weight of the reference by
- adding a filler.
- The filler can include every material or composition having a higher density than the reference like for example oxides, carbonates, sulfates and silicates.
- 3) Take one of the SPCs or Additive Concentrates as a reference and increase the specific weight of the SPCs or Additive Concentrates with lower specific weight to the specific weight of the reference and reduce the specific weight of the SPCs or Additive Concentrates with higher specific weight to the specific weight of the reference as described above.
- Antistatic agents are added, when necessary, to the SPCs and Additive Concentrates to prevent the de-mixing of the mixture, respectively the blend, due to electrostatic charge of the SPCs or Additive Concentrates.
- All the tests performed in the examples show that the mixture of different SPCs and or Additive Concentrates with equal density according to the invention, stay homogeneously mixed, e.g. when vibrated with a back and forth shaker, compared to a mixture of SPCs with non-equal density.

Examples

The following SPCs have been prepared as described:

- 5 A) White P.W. 6: 28% of TiO_2 has been mixed with 72% of polypropylene and the mixture has been dispersed with a twin screw extruder.
- B) White 2 P.W. 6: 70% of TiO_2 has been mixed with 30% of polypropylene and the mixture has been dispersed with a twin screw extruder.
- 10 C) Yellow P.Y. 34 : 27% of Chrome Yellow has been mixed with 63% of polypropylene and the mixture has been dispersed with a twin screw extruder
- D) Yellow P.Y. 119 : 25% of Zinc/Ferric oxide has been mixed with 75% of polypropylene and the mixture has been dispersed with a twin screw extruder
- E) Black P.Bk. 7: 35% of Carbon black has been mixed with 65% of polypropylene and the mixture has been dispersed with a twin screw extruder
-

15

The bulk density of these SPCs has then been adjusted to $0.62 \pm 0.02 \text{ g/cm}^3$ according to ISO 697 by adjusting the concentration of the pigment dispersed in the polymer carrier.

20 And

- F) Red PR 214: 40% of PV Fast Red BNP has been mixed with 60% of polypropylene and the mixture has been dispersed with a twin screw extruder

The bulk density of this SPCs has not been adjusted

25

The following Additive Concentrate has been prepared as described:

- 30 G) Anti-oxidant Irganox[®] B501W (Trademark of Ciba Specialities Inc.): 15% of Irganox B501W has been mixed with 30% of calcium carbonate and 55% of polypropylene and the mixture has been dispersed with a twin screw extruder.

The bulk density of this Additive Concentrate Blend has been adjusted to $0.62 \pm 0.02 \text{ g/cm}^3$ according to ISO 697 by adding a filler (calcium carbonate).

| | Component (SPC or Additive Concentrate) | Bulk Density (g/cm ³) |
|---|---|-----------------------------------|
| A | White P.W. 6 | 0.60 – 0.62 |
| B | White 2 P.W. 6 | 1.04 – 1.06 |
| C | Yellow PY 34 | 0.60 – 0.62 |
| D | Yellow PY 119 | 0.61 – 0.63 |
| E | Black P.Bk. 7 | 0.60 – 0.62 |
| F | Red P.R. 214 | 0.62 – 0.64 |
| G | Irganox [®] B501W | 0.62 – 0.64 |

Examples 1-4

5 The following batches have been prepared:

Batch 1

9.98% Irganox[®] B501W

78.63% White P.W. 6

10 4.27% Yellow P.Y. 34

6.30% Yellow P.Y. 119

0.82% Black P.Bk. 7

Total batch size: 50 g + 0.025 g Nopcostat[®] HS (Antistatic) (Trademark of Cognis)

15 **Batch 2**

6.63% Irganox[®] B501W

31.84% White P.W. 6

56.52% Yellow P.Y. 34

2.95% Red P.R. 242

20 2.06% Black P.Bk. 7

Total batch size: 50 g + 0.025 g Nopcostat[®] HS (Antistatic) (Trademark of Cognis)

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Batch 3

9.98% Irganox® B501W
78.63% White P.W. 6
4.27% Yellow P.Y. 34
5 6.30% Yellow P.Y. 119
0.82% Black P.Bk. 7

Total batch size : 50 g

Batch 4

10 6.63% Irganox® B501W
31.84% White P.W. 6
56.52% Yellow P.Y. 34
2.95% Red P.R. 242
2.06% Black P.Bk. 7

15 Total batch size: 50 g

The following tests have been carried out:

The batches 1 to 4 have been agitated with a back and forth shaker from Infors AG at
20 the speed of 400 rpm for 24 hours.

Batches 1 and 2 (with antistatic agent) stay substantially homogeneous whereas the
batches 3 and 4 (without antistatic agent) show a significantly higher de-mixing
tendency due to electrostatic charging.

25

Comparative Examples 5-8

----- The following 3 batches have been prepared by filling two separate layers in a bottle: -----

5 Batch 5

Upper layer: 50.0% White 2 P.W. 6

Bottom layer: 50.0% Black P.Bk. 7

Total batch size: 50 g

10

Batch 6

Upper layer: 50.0% Black P.Bk. 7

Bottom layer: 50.0% White 2 P.W. 6

15 Total batch size: 50 g

Batch 7

Upper layer: 50.0% White P.W. 6

Bottom layer: 50.0% Black P.Bk. 7

20

Total batch size: 50 g

The following batch has been prepared by filling a random mixture in a bottle:

25

Batch 8

50.0% White P.W. 6

50.0% Black P.Bk. 7

30 Total batch size: 50 g

The following tests have been carried out:

The Batches 5 to 8 have been agitated with a back and forth shaker from Infors AG at the speed of 400 rpm for 24 hours.

5

The two layers of Batch 5 were completely mixed due to the difference of bulk densities, whereas in Batch 6 only a few black granules arrived in the white layer. (In Batch 6 the higher density of the white component prevents the pellets from moving upward whereas the lower density of the black component prevents the pellets from moving downward.)

10

The two layers of Batch 7 stayed substantially separate due to the equal bulk density. (Only a few black and white granules arrived in the opposite layer.)

15 In the random mixture of Batch 8 no de-mixing could be observed due to the equal bulk density.

Claims

-
1. Pellets or micro pellets of one or more Single Pigment Concentrates (SPCs) and
5 of one or more Additive Concentrates with at least two different chemical
compositions,
or of two or more Single Pigment Concentrates (SPCs) with at least two different
chemical compositions,
10 or of two or more Additive Concentrates with at least two different chemical
compositions,
characterised in that the pellets or micro pellets with different chemical
compositions are of substantially equal density.
-
- 15 2. Pellets or micro pellets according to claim 1, characterised in that they are of
substantially equal size.
- 20 3. Pellets or micro pellets according to claims 1 or 2, characterised in that they are of
substantially equal shape.
- 25 4. Pellets or micro pellets according to claims 1 to 3, characterised in that they have
substantially equal electrostatic properties.
- 30 5. Pellets or micro pellets according to claims 1 to 4, characterised in that the
particle size of the pellets is from larger than 2.0 mm to 4.0 mm and that the
particle size of the micro pellets is from 0.5 to 2.0 mm.
6. Pellets or micro pellets according to claims 1 to 5, characterised in that the density
of the pellets is from 0.5 to 1.5 g/cm³, preferably from 0.6 to 1.1 g/cm³.
-

7. Masterbatch comprising pellets or micro pellets of one or more Single Pigment Concentrates (SPCs) and of one or more Additive Concentrates according to any one of claims 1 to 6.
- 5 8. Process for the preparation of pellets or micro pellets according to claims 1 to 6, characterised in that the Single Pigment Concentrate (SPC) or Additive Concentrate with the chemical composition of lower specific weight is taken as reference and that the specific weight of all other SPCs or Additive Concentrates is reduced to the specific weight of the reference by reducing the pigment
10 concentration of the SPCs or additive concentration of the Additive Concentrates.
9. Process for the preparation of pellets or micro pellets according to claims 1 to 6, characterised in that the Single Pigment Concentrate (SPC) or Additive
15 ~~Concentrate with the chemical composition of lower specific weight is taken as~~ reference and that the specific weight of all other SPCs or Additive Concentrates is reduced to the specific weight of the reference by adding a blowing agent to the SPCs or Additive Concentrates.
- 20 10. Process for the preparation of pellets or micro pellets according to claims 1 to 6, characterised in that the Single Pigment Concentrate (SPC) or Additive Concentrate with the chemical composition of higher specific weight is taken as reference and that the specific weight of all other SPCs or Additive Concentrates is increased to the specific weight of the reference by adding a filler to the SPCs
25 or Additive Concentrates.
11. Use of the pellets or micro pellets according to claims 1 to 6 for the manufacture of Masterbatches.
- 30 12. Use of Masterbatches according to claim 7 for bringing color and additive properties in to plastics.

Abstract

The invention relates masterbatches comprising pellets or micro pellets of one or more
5 Single Pigment Concentrates (SPCs) and of one or more Additive Concentrates with at
least two different chemical compositions, or of two or more Single Pigment
Concentrates (SPCs) with at least two different chemical compositions, or of two or
more Additive Concentrates with at least two different chemical compositions,
characterised in that the pellets or micro pellets with different chemical compositions
10 are of substantially equal density, size, shape and/or electrostatic properties.

The invention further relates to a process for preparing said pellets or micro pellets in
that a Single Pigment Concentrate (SPC) or Additive Concentrate with a chemical
~~composition of specific weight is taken as reference and that the specific weight of all~~
15 other SPCs or Additive Concentrates is adjusted to the specific weight of the reference
by adapting the concentration of pigment (in the SPC) or additive (in the Additive
concentrate) or by adding a filler or a blowing agent to the SPCs or Additive
Concentrates.

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